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THE EFFECT OF SMELTER ATMOSPHERES ON THE QUALITY OF DRY PROCESS ENAMELS FOR CAST IRON

A REPORT OF AN INVESTIGATION

CONDUCTED BY

THE ENGINEERING EXPERIMENT STATION
UNIVERSITY OF ILLINOIS

IN COÖPERATION WITH

THE UTILITIES RESEARCH COMMISSION

BY

ANDREW I. ANDREWS

AND

H. WILBUR ALEXANDER

BULLETIN No. 227

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THE EFFECT OF SMELTER ATMOSPHERES ON THE QUALITY OF DRY PROCESS ENAMELS FOR CAST IRON

I. Introduction

1. *Purpose of Investigation.*—The effect of various gases present in the furnace during the smelting of enamels for sheet steel has been covered in Bulletin No. 224 recently published.* It was concluded that nitrogen, carbon dioxide, and reducing atmospheres, determined by the presence of CO and the lack of oxygen, were not deleterious to enamels for sheet steel and that sulphur dioxide was deleterious. It was the purpose of this investigation to continue this work on another type of enamels, dry process enamels for cast iron. It was decided to subject these dry process enamels to the following smelter atmospheres: (1) sulphur dioxide, (2) sulphur dioxide and reducing conditions, and (3) reducing conditions. The effect of these atmospheres on the quality of the enamel after it had been burned on cast-iron test pieces was determined.

2. *Acknowledgments.*—The data presented in this paper were obtained in an investigation which was conducted by the Engineering Experiment Station of the University of Illinois, of which Dean M. S. Ketchum is the director, in coöperation with the Utilities Research Commission† which represents several Utilities Companies, chiefly in Chicago, Illinois. This research was carried out in the Department of Ceramic Engineering of which Prof. C. W. Parmelee is the head.

The writers wish to thank Professor Parmelee for his helpful suggestions and coöperation in this research.

II. The Smelting Equipment

3. *Electric Smelting Equipment.*—The Globar electric laboratory smelter described in Bulletin No. 224 was used in this work. The only addition to the smelting equipment was a small exhaust hood p aced

*"The Effect of Smelter Atmospheres on the Quality of Enamels for Sheet Steel," Univ. of Ill. Eng. Exp. Sta. Bul. 224.

†The Utilities Research Commission represents Chicago, North Shore & Milwaukee Railroad Company, Public Service Company of Northern Illinois, The People's Gas Light & Coke Company, Chicago Rapid Transit Company, Commonwealth Edison Company, Middle West Utilities Company, and Midland United Company.

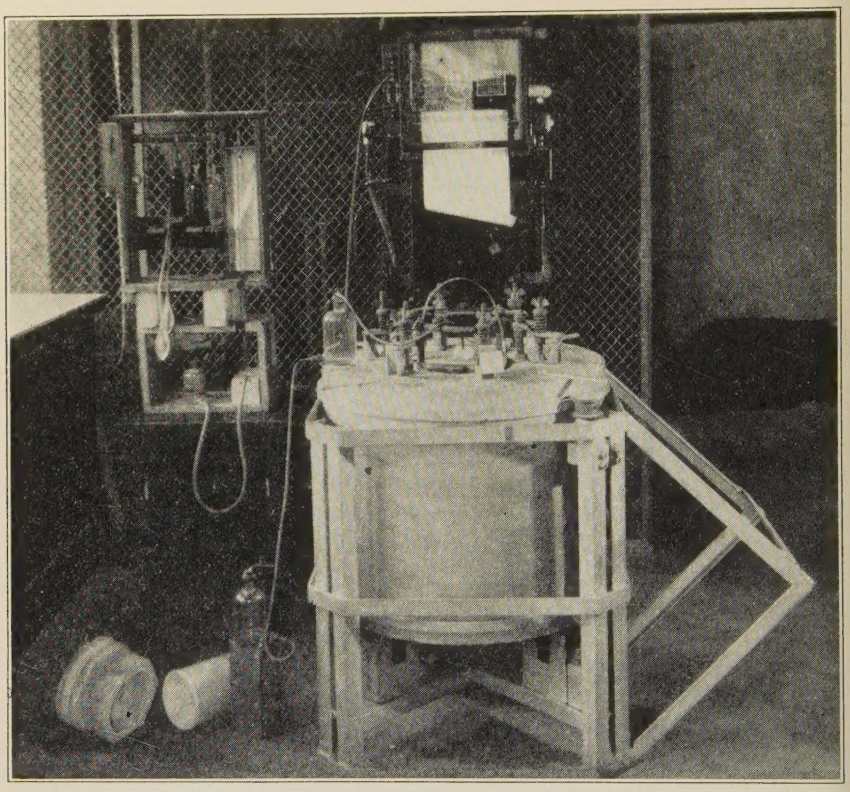

FIG. 1. ELECTRIC LABORATORY FURNACE

over the smelter lid to catch escaping gases which might be harmful to the investigators and to delicate laboratory equipment.

III. DETERMINATION OF EFFECT OF SMELTER ATMOSPHERES ON QUALITY OF DRY PROCESS ENAMELS FOR CAST IRON

4. *Enamels Used.*—The compositions of the enamels used in this work are given in Table 1. R42, a leadless tin enamel, R46 a high-lead tin enamel, R49, a low-lead antimony enamel, and R52, a leadless antimony enamel, were smelted in one and one-half hours at a furnace temperature of 2025 deg. F. giving a temperature of 1860 deg. F. over the molten enamel at the end of the test. N6, a low-lead antimony enamel containing TiO_2, was more refractory and required a furnace temperature of 2300 deg. F., giving a temperature of about 2100 deg.

TABLE 1
DRY PROCESS ENAMELS FOR CAST IRON*

	R42	R46	R49	R52	N6
Feldspar	33.4	33.1	32.0	33.2	8.5
Borax	19.6	18.7	24.7	28.2	11.9
Flint	....	....	....	....	25.7
Na_2CO_3	....	2.6	3.7	....	14.4
$NaNO_3$	2.4	2.6	2.9	5.6	5.1
Fluorspar	....	4.4	6.0	4.8	....
Cryolite	9.7	2.6	....	3.2	....
Red Lead	....	14.6	2.6	....	7.6
ZnO	10.2	9.2	9.1	11.3	5.1
Boric Acid	17.9	....	....	....	....
$BaCO_3$	....	4.4	6.6	6.5	....
Whiting	....	....	2.5	2.4	3.0
Na_2SiF_6	....	....	....	....	1.7
Sb_2O_3	....	....	....	4.8	....
Sodium Antimonate	....	....	9.9	....	10.2
SnO_2	6.8	7.8	....	....	....
TiO_2	....	....	....	....	6.8
Total	100.0	100.0	100.0	100.0	100.0

*R42, R46, R49, R52—Tech. Paper, No. 142, Bureau of Standards, H. F. Staley.
N6—"Acid Resisting Cover Enamels for Sheet Iron," Univ. of Ill. Eng. Exp. Sta. Bul. 201.

F. over the molten enamel at the end of the test. One and one-half hours were required for complete fusion.

5. *Method of Procedure and Analysis.*—A 1000-gram batch of each enamel was thoroughly mixed and, when the smelter had reached the desired temperature, the frit batch was placed in the crucible through a hole in the lid by means of a funnel. The test gases were introduced through holes in the lid of the crucible, city gas of the following composition being used to give reducing conditions:

	Per Cent		*Per Cent*
CO_2	4.8	H_2	28.1
Illuminants	10.4	CH_4	15.5
O_2	0.8	N_2	18.6
CO	21.8	S	6-9 grains per 100 cu. ft.

Sampling tubes and gas tubes were sealed in the lid with a mixture of asbestos, sodium silicate, and water. It was found that one and one-half hours smelting in air was sufficient to give a good smooth thread, which was the criterion for complete fusion. The molten frit was quenched in cold water, rinsed, dried, ground to 100 mesh, and burned on cast-iron test pieces in air, the effect of the smelting atmospheres being determined from these test pieces. The atmospheres over the enamel during smelting were analyzed at suitable intervals for carbon dioxide, oxygen, and carbon monoxide, the carbon dioxide being absorbed in a potassium hydroxide solution, the oxygen in an

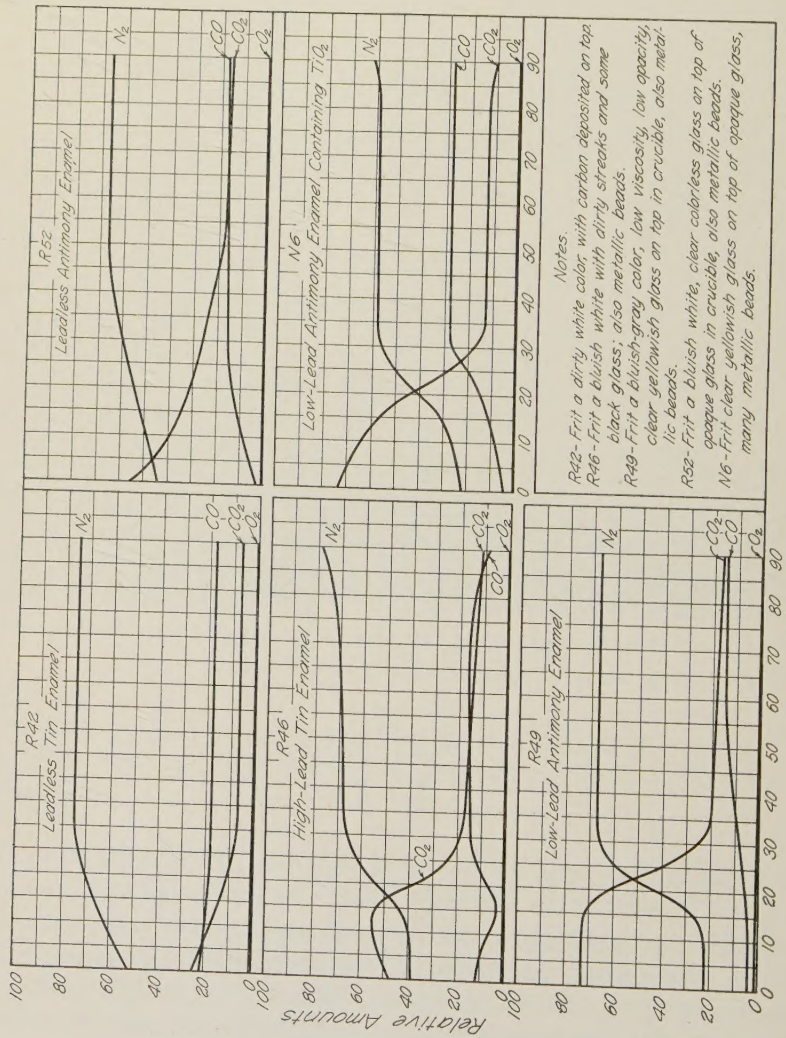
R42
Leadless Tin Enamel
R52
Leadless Antimony Enamel
R46
High-Lead Tin Enamel
N6
Low-Lead Antimony Enamel Containing TiO2
R49
Low-Lead Antimony Enamel
N2
CO
CO2
O2
Relative Amounts
Time in Minutes after Start of Test
Notes:
R42- Frit a dirty white color, with carbon deposited on top
R46- Frit a bluish white with dirty streaks and some black glass; also metallic beads.
R49- Frit a bluish-gray color, low viscosity, low opacity, clear yellowish gloss on top in crucible, also metallic beads.
R52- Frit a bluish white, clear colorless glass on top of opaque glass in crucible, also metallic beads.
N6- Frit clear yellowish glass on top of opaque glass, many metallic beads.

FIG. 2. FURNACE ATMOSPHERE ANALYSES UNDER REDUCING CONDITIONS WITH DRY PROCESS ENAMELS FOR CAST IRON

alkaline solution of pyrogallol, and the carbon monoxide in an acid solution of cuprous chloride. The water soluble gases were dissolved in the salt water of the gas collecting bottles before making the analysis. This procedure removed the fluorides, probably most of the nitrogen oxides, and any sulphur dioxide present. The gases were allowed to cool before analyzing. For sulphur dioxide the method of Raschig as given by Dennis* was used. This is a modification of the iodine method in which sodium acetate solution is added to prevent the interference of nitrous acid. A blank test made with both enamels used in the tests on sheet steel, Bulletin No. 224, showed that any interference by substances other than nitrogen oxides evolved from the enamel was negligible. In fact, the tests proved that even the regular Reich iodine method was satisfactory under the given conditions.

Since sulphur dioxide reacts with the enamel to form sulphates which may be soluble, and thus could be removed by careful washing, tests were made to determine the effect of quenching and washing on the quality of enamels smelted in atmospheres high in sulphur dioxide. The following procedure was used:

The regular smelting procedure was followed. The molten frit was quenched in 10 000 cc. of distilled water and allowed to remain in the water for a total of 4 minutes, then it was rinsed with 500 cc. of the quenching water. Two samples of 500 cc. each were then taken and the frit was replaced in the water and kept well agitated for 11 minutes more, and two more 500 cc. samples were taken. The four samples of quenching water were analyzed for soluble sulphates that had dissolved out of the frit. Each sample was filtered, made slightly acid with HCl using methyl orange as an indicator, and the sulphate was precipitated as $BaSO_4$, filtered and dried, heated to redness, cooled and weighed. The sulphate was then calculated to K_2SO_4, or Na_2SO_4. The chemical analysis of the sulphate formed, given in Bulletin No. 224, shows it to be a combination of K_2SO_4 and Na_2SO_4. An examination with X-rays by the Hull-Debye-Scherrer powder method indicates that neither K_2SO_4 nor Na_2SO_4 are present alone, but that some combination of the two is present. The X-ray tests were made possible through the courtesy of Dr. G. L. Clark of the Department of Chemistry of the University of Illinois.

6. *Effect of Reducing Conditions on Dry Process Enamels for Cast Iron.*—The analyses of the atmospheres are shown in Fig. 2. Because of the volume of gas given off by the enamel, the atmosphere

*L. M. Dennis, "Gas Analysis," The Macmillan Company.

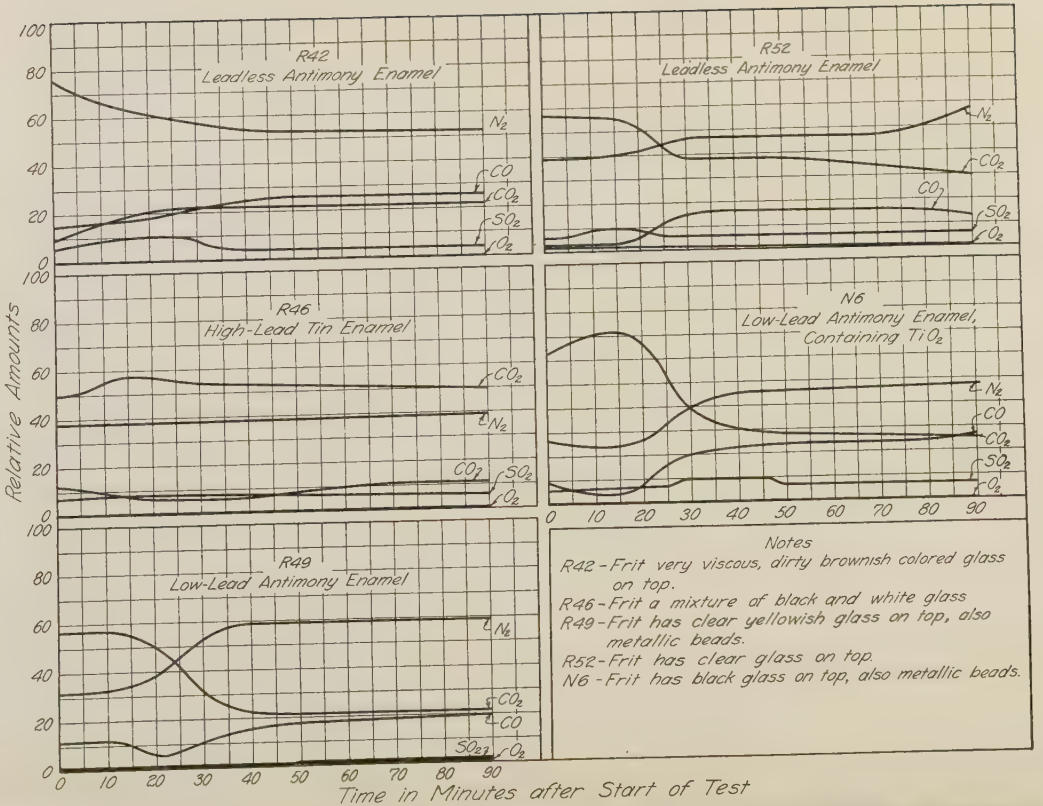

FIG. 3. FURNACE ATMOSPHERE ANALYSES UNDER SO_2 PLUS REDUCING CONDITIONS WITH DRY PROCESS ENAMELS FOR CAST IRON

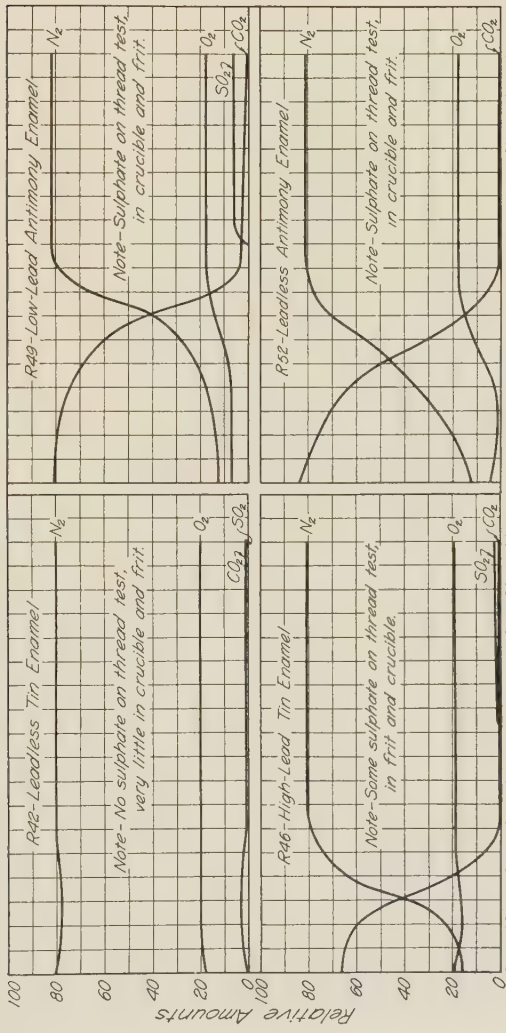
R49-Low-Lead Antimony Enamel
Note-Sulphate on thread test, in crucible and frit
R52-Leadless Antimony Enamel
Note-Sulphate on thread test, in crucible and frit
R42-Leadless Tin Enamel
Note-No sulphate on thread test, very little in crucible and frit
R46-High-Lead Tin Enamel
Note-Some sulphate on thread test, in frit and crucible
N_2
O_2
SO_2
CO_2
Relative Amounts
Time in Minutes after Start of Test

FIG. 4. FURNACE ATMOSPHERE ANALYSES UNDER LOW SO_2 CONCENTRATIONS WITH DRY PROCESS ENAMELS FOR CAST IRON

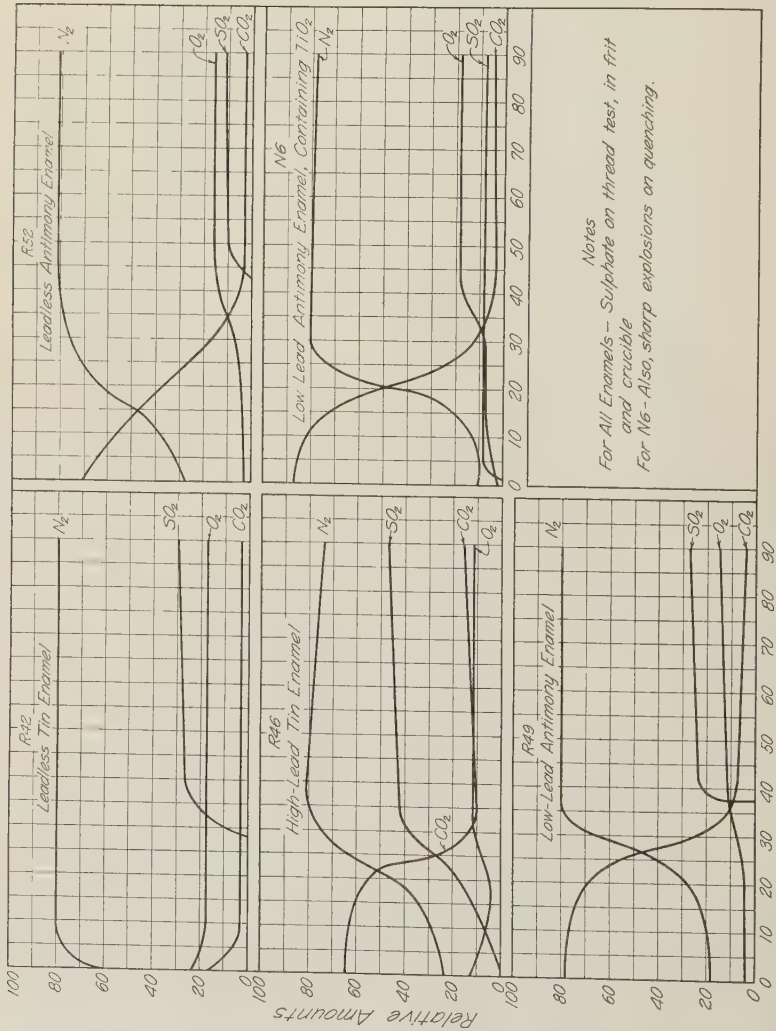

FIG. 5. FURNACE ATMOSPHERE ANALYSES UNDER HIGHER SO_2 CONCENTRATIONS WITH DRY PROCESS ENAMELS FOR CAST IRON

did not become approximately constant until the test was half completed. R42, a leadless tin enamel, was the only one to withstand the reducing action of the city gas. In all other cases metal oxides were reduced to metallic beads. It is presumed that the oxides reduced are the opacifying oxides, as there was a decided loss in opacity in some cases, and the appearance of white refractory oxidized particles in the surface of the enamel in other cases. In some cases the loss of opacity was so great as to give a mottled appearance to the enamel. A qualitative test of the metallic beads formed in R49, a low-lead antimony enamel, showed only antimony to be present. A qualitative test of the metallic beads formed in R46, a high-lead tin enamel, showed only the presence of metallic tin. With N6, the low-lead antimony enamel containing TiO_2, the reduction was so great that the enamel would not burn down in the ordinary burning time.

7. *Effect of Reducing Conditions and Sulphur Dioxide on Dry Process Enamels for Cast Iron.*—The analyses of the atmospheres are shown in Fig. 3. Reducing conditions prevent the formation of sulphates in the enamel by the sulphur dioxide. Because of the reduction of part of the sulphur dioxide to hydrogen sulphide by the reducing atmosphere, a lower sulphur dioxide content was thus recorded than in the tests with sulphur dioxide alone. The amount of sulphur dioxide entering the smelting chambers was approximately the same for all tests.

In all cases the SO_2- reducing atmospheres were deleterious to the quality of the enamel. The leadless enamels, R42 and R52, were most resistant, showing only dirty surfaces, while R49 and N6 had refractory oxides appearing and a mottled opacity. R46 was least resistant, being changed from a good white enamel to a glossy black enamel with evenly distributed fine white specks.

8. *Effect of Sulphur Dioxide on Dry Process Enamels for Cast Iron and Relation of Quenching Practice to the Effect.*—The analyses of the atmospheres of the first tests made are shown in Fig. 4. The results obtained indicate that even when the frit is allowed to remain in the quenching water four minutes and then rinsed once, approximately five per cent sulphur dioxide in the atmosphere at the end of the test will cause scumming and sulphur pock marks. Tests were then made with greater amounts of sulphur dioxide in the atmosphere (analyses shown in Fig. 5), to determine the effect of the higher concentrations of sulphur dioxide and the relation of quenching practice to the effect. In all cases, except R46, the 15-minute quenching

TABLE 2

EFFECT OF SULPHUR DIOXIDE ON DRY PROCESS ENAMELS FOR CAST IRON AND RELATION OF QUENCHING PRACTICE TO THE EFFECT

(A) Low Percentages of SO_2, 4-min. Quenching, No Soluble Sulphate Test

Enamel	Per Cent SO_2 in Atmosphere	Burning Quality
R42	0.02–0.56	Good surface
R46	0.05–2.51	Good surface
R49	0.04–6.18	S pock marks
R52	0.00–0.01	Good surface

(B) High Percentages of SO_2, Soluble Sulphate Test

Enamel	Per Cent SO_2 in Atmosphere	Per Cent Sulphate Removed from Frit				Burning Quality
		4 min. test calculated as K_2SO_4 or Na_2SO_4		15 min. test calculated as K_2SO_4 or Na_2SO_4		
R42	0.28–30.1	0.96	0.78	1.11	0.91	Good surface
R46	1.56–46.2	2.89	2.35	3.21	2.61	S pock marks
R49	0.05–3.24	1.40	1.14	1.62	1.32	Good surface
R49	0.05–27.7	2.00	1.63	2.54	2.07	Good surface
R52	0.03–11.25	1.79	1.46	1.93	1.57	Good surface
N6	2.15–8.67	3.06	2.49	3.42	2.78	Good surface

and washing period was sufficient to remove the soluble sulphates formed during smelting and give a good enamel. The atmosphere of R46 was exceedingly high in sulphur dioxide, and the percentage of soluble sulphates in the frit was higher than in the other enamels. A longer and more careful quenching and washing may be required for very high concentrations of sulphur dioxide. Table 2 shows the percentage of soluble sulphates.

IV. SUMMARY OF RESULTS AND GENERAL CONCLUSIONS

9. *Summary of Results.*—Following is a summary of the results obtained from the investigation:

(1) Reducing atmospheres in the smelter were deleterious to all the enamels tested, except the leadless tin enamel, causing the reduction of metal oxides to metallic beads, which resulted in a loss of opacity, small spots of refractory oxides in the surface, and black specks on burning.

(2) Reducing atmospheres and the presence of high percentages of sulphur dioxide in the smelter were deleterious to all enamels tested, causing the reduction of metal oxides to metallic beads, which re-

sulted in a loss of opacity, small spots of refractory oxides in the surface, and black specks on burning. The leadless tin enamel, however, was least affected.

(3) The lead enamels, R46, R49, N6, smelted under reducing conditions and under reducing conditions in the presence of sulphur dioxide, had a mottled appearance, and R46 (SO_2- reducing) was altered from a good white enamel to a glossy black enamel with evenly distributed fine white specks.

(4) Enamels R42, R46, R52, smelted in atmospheres low in sulphur dioxide, containing 2.5 per cent SO_2 or less at the end of the test, and quenched for four minutes, were of good quality on burning.

(5) The enamel R49, smelted in an atmosphere containing 6.2 per cent sulphur dioxide at the end of the test, and quenched for four minutes, showed the characteristic sulphur pock marks on burning, due to incomplete removal of the sulphate.

(6) Enamels smelted in atmospheres high in sulphur dioxide, containing up to 30 per cent at the end of the test, and quenched and washed for fifteen minutes were of good quality on burning.

(7) The enamel R46, smelted in an atmosphere containing 46 per cent sulphur dioxide at the end of the test, showed the characteristic sulphur pock marks with the fifteen-minute quenching and washing period. Apparently, the washing was incomplete.

(8) The sulphur dioxide apparently reacted more readily with the enamel during the first half of the smelting period, or the period during which the greatest volumes of gases were being given off.

(9) With N6, reducing conditions caused an increase in refractoriness, as well as loss of opacity, refractory oxides in surface, and black specks.

10. *General Conclusions.*—The conclusions drawn are as follows:

(1) Composition plays an important part in the resistance of dry process enamels for cast iron to the effect of reducing smelter atmospheres and reducing smelter atmospheres with sulphur dioxide present.

(a) The leadless tin enamel is the most resistant to the reducing conditions.

(b) The high-lead enamel is less resistant to reducing conditions and reducing conditions with sulphur dioxide present than the low-lead enamels tested.

(c) It should be possible to develop leadless enamels that would be resistant to any reducing conditions found in plant smelting practice.

(d) Tin oxide is less readily reduced than antimony oxide.

(2) Careful quenching and washing will remove the sulphate formed by smelting in the presence of high percentages of sulphur dioxide, and no scumming or sulphur pock marks will result from that source.

(a) Any sulphate formed from the sulphur dioxide in fuels used in regular plant smelting practice should be removed by the regular water quenching practice.

(b) Careful quenching and washing are advisable in all cases.

(3) Since, in this investigation, only gases in high concentrations caused defects to appear, it is very probable that many of the black specks encountered in commercial smelting are due to carbon or other foreign materials being trapped in the enamel.

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